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Morusin and Morusin Dimethyl Ether

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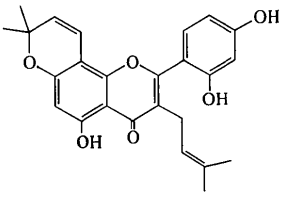
Abstract

The structures of morusin, 2-(2,4-dihydroxyphenyl)-5-hydroxy-8,8-dimethyl-3-(3-methyl-2-butenyl)-4*H*,8*H*-benzo[1,2-*b*;3,4-*b*']dipyran-4-one, $C_{25}H_{24}O_6$ (I), and morusin dimethyl ether, 2-(2,4-dimethoxyphenyl)-5-hydroxy-8,8-dimethyl-3-(3-methyl-2-butenyl)-4*H*,8*H*-benzo[1,2-*b*;3,4-*b*']dipyran-4-one, $C_{27}H_{28}O_6$ (II), are reported. In compounds (I) and (II), the benzopyranone moiety is planar. The dihedral angles between the benzopyranone moiety and the phenyl ring are 66.25 (10) and 85.32 (5)°, respectively. When the dihydroxyphenyl [dimethoxyphenyl for (II)] and 3-methyl-2-butenyl groups are fixed in the same positions to the benzopyranone plane, the pyrano rings of (I) and (II) are enantiomeric.

Comment

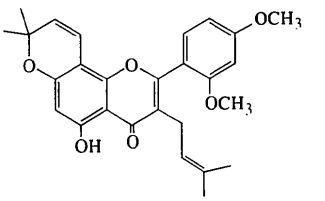
The photoreaction of the title compound [morusin: (I)] with a high-pressure mercury lamp gives the hydroperoxide with an ether linkage between an alkenyl group



(I)

at C3 and an hydroxyl group at C2′ (Nomura, Fukai, Yamada & Katayanagi, 1977). Crystals of morusin (I) and its dimethyl ether (II) have been prepared so that the interaction between the above two functional groups participating in the formation of the ether linkage can be examined by X-ray diffraction.

The molecular structures of (I) and (II) are shown in Figs. 1 and 2, respectively. In both (I) and (II), the benzopyranone moieties are planar. The dihedral angles between the planes O1,C2,C3,C4,C10,C9 and C5,C6,C7,C8,C9,C10 are 1.7 (1) and 1.7 (2)° for (I)



(II)

and (II), respectively. The dihydroxyphenyl [dimethoxyphenyl for (II)] and 3-methyl-2-butenyl groups are not coplanar with the benzopyranone in either molecule; the torsion angles C3—C2—C1′—C2′ and C4—C3—C11—C12 are 67.9 (4) and 74.3 (4), and 85.5 (2) and 96.4 (2)° in (I) and (II), respectively. The former values are comparable to those of 62.2 (1)° in 6-hydroxy-2′,3′-dimethoxyflavone (Wallet, Gaydou, Mas, Molins & Miravitlles, 1993) and 68.2° in 7-hydroxy-2′,6′-dimethoxyflavone (Wallet, Gaydou, Espinosa, Osorno, Molins & Miravitlles, 1992). One of the two methoxy groups in (II) is coplanar with the phenyl ring, as shown by the C5′—C4′—O4′—C8′ torsion angle of −1.3 (3)°. The other is twisted slightly out of the plane [C3′—C2′—O2′—C7′ = 10.9 (3)°]; this can be attributed to the steric hindrance between the methoxy and 3-methyl-2-butenyl groups. The puckering of the 1,3-diplanar pyrano rings of (I) and (II) is mirror related. This can be seen if the relative positions of the dihydroxyphenyl [dimethoxyphenyl for (II)] and 3-methyl-2-butenyl groups to the benzopyranone group are defined by torsion angles, *e.g.* O1—C2—C1′—C2′ and C4—C3—C11—C12, with the same signs (−− and ++). Then the deviations of O7 and C16 from the C7,C8,C18,C17 plane are −0.164 (3) and 0.346 (7) Å in (I); the corresponding values for (II) are 0.163 (3) and −0.291 (4) Å, respectively.

In both molecules, intramolecular hydrogen bonding occurs between the hydroxyl- and ketonic-O atoms of the benzopyranone group. In the Figs. 1 and 2, the H

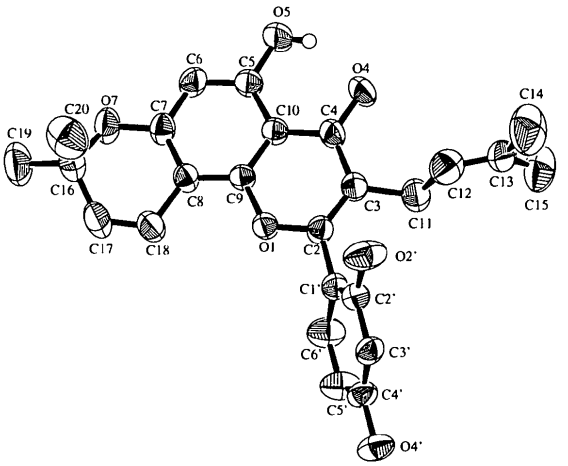


Fig. 1. The perspective drawing of morusin. Displacement ellipsoids are drawn at the 50% probability level.

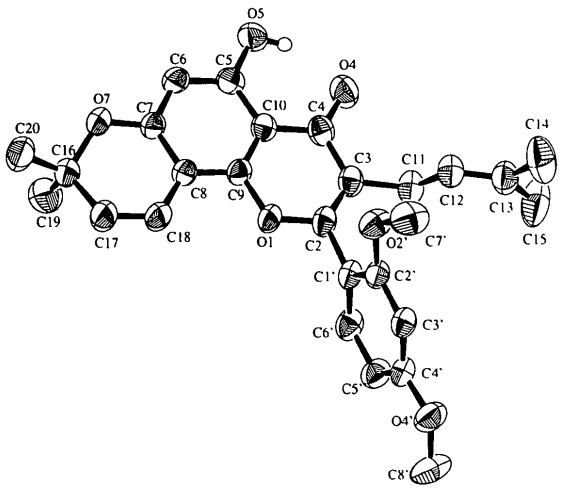


Fig. 2. The perspective drawing of morusin dimethyl ether. Displacement ellipsoids are drawn at the 50% probability level.

atom bonded to O5 has been included. The O4···O5 distances of 2.601 (3) and 2.581 (2) Å for (I) and (II), respectively, compare with the values of 2.612 (5) Å reported for hymanoxin (Watson, Kashyap, Gao & Mabry, 1991) and 2.580 (2) Å reported for centaureidin (Fronczek, Parodi & Fischer, 1989).

Experimental

Compound (I) was isolated from *Morus alba Linne* and (II) was synthesized by the etherification of morusin

Compound (I)

Crystal data

$C_{25}H_{24}O_6$
M_r = 420.44
Monoclinic
$P2_1/c$
a = 11.0672 (8) Å
b = 17.6850 (12) Å
c = 12.0475 (8) Å
β = 111.945 (5)°
V = 2187.1 (3) Å^3
Z = 4
D_x = 1.277 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.54180 Å
Cell parameters from 25 reflections
θ = 57–60°
μ = 0.748 mm^{-1}
T = 293 (2) K
Prism
0.4 × 0.4 × 0.2 mm
Colourless

Data collection

Rigaku AFC-6 four-circle diffractometer
Profile data from ω–2θ scans
Absorption correction: none
3463 measured reflections
3463 independent reflections
2639 observed reflections [$I > 2\sigma(I)$]

θ_{max} = 62.49°
h = 0 → 12
k = 0 → 20
l = −13 → 12
3 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0605
$wR(F^2)$ = 0.1848
S = 0.778
3463 reflections
287 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1678P)^2 + 2.4812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.022
$\Delta\rho_{max}$ = 0.601 e Å^{-3}
$\Delta\rho_{min}$ = −0.274 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.3324 (2)	0.13461 (10)	−0.0365 (2)	0.0452 (5)
C2	0.2230 (3)	0.1162 (2)	−0.1318 (2)	0.0449 (7)
C3	0.1721 (3)	0.0462 (2)	−0.1496 (3)	0.0518 (8)
C4	0.2310 (3)	−0.0118 (2)	−0.0608 (3)	0.0502 (7)
O4	0.1835 (2)	−0.07745 (12)	−0.0695 (2)	0.0690 (7)
C5	0.4215 (3)	−0.0448 (2)	0.1248 (3)	0.0516 (8)
O5	0.3826 (3)	−0.11763 (12)	0.1187 (2)	0.0754 (8)
C6	0.5313 (3)	−0.0221 (2)	0.2198 (3)	0.0547 (8)
C7	0.5693 (3)	0.0531 (2)	0.2279 (3)	0.0487 (7)
C8	0.5060 (3)	0.1064 (2)	0.1415 (3)	0.0462 (7)
C9	0.3960 (3)	0.08153 (15)	0.0469 (2)	0.0416 (6)
C10	0.3493 (3)	0.0075 (2)	0.0364 (2)	0.0437 (7)
C1′	0.1750 (3)	0.1828 (2)	−0.2104 (3)	0.0470 (7)
C2′	0.0565 (3)	0.2177 (2)	−0.2267 (3)	0.0499 (7)
O2′	−0.0244 (3)	0.1940 (2)	−0.1727 (3)	0.0844 (9)
C3′	0.0143 (3)	0.2795 (2)	−0.3020 (3)	0.0513 (7)
C4′	0.0890 (3)	0.3066 (2)	−0.3609 (3)	0.0515 (8)
O4′	0.0507 (3)	0.36732 (12)	−0.4381 (2)	0.0647 (7)
C5′	0.2078 (4)	0.2736 (2)	−0.3455 (4)	0.0721 (10)
C6′	0.2503 (4)	0.2132 (2)	−0.2690 (3)	0.0671 (10)
C11	0.0657 (3)	0.0245 (2)	−0.2659 (3)	0.0615 (9)
C12	−0.0619 (4)	0.0082 (2)	−0.2591 (4)	0.0739 (10)
C13	−0.1596 (3)	−0.0314 (2)	−0.3411 (3)	0.0688 (10)
C14	−0.2853 (5)	−0.0400 (4)	−0.3265 (5)	0.121 (2)
C15	−0.1539 (4)	−0.0658 (3)	−0.4485 (4)	0.101 (2)
O7	0.6775 (2)	0.07265 (13)	0.3238 (2)	0.0633 (6)
C16	0.6929 (3)	0.1526 (2)	0.3620 (3)	0.0652 (9)
C17	0.6512 (3)	0.2030 (2)	0.2552 (3)	0.0675 (10)
C18	0.5600 (3)	0.1823 (2)	0.1533 (3)	0.0594 (8)
C19	0.8365 (4)	0.1595 (3)	0.4358 (5)	0.100 (2)
C20	0.6091 (5)	0.1653 (3)	0.4342 (5)	0.102 (2)

Table 2. *Selected geometric parameters (Å, °) for (I)*

O1—C9	1.363 (3)	C1′—C6′	1.386 (4)
O1—C2	1.360 (3)	C1′—C2′	1.395 (4)
C2—C3	1.344 (4)	C2′—O2′	1.355 (4)
C2—C1′	1.480 (4)	C2′—C3′	1.385 (4)
C3—C4	1.448 (4)	C3′—C4′	1.363 (4)
C3—C11	1.505 (4)	C4′—O4′	1.379 (4)
C4—O4	1.262 (4)	C4′—C5′	1.386 (5)
C4—C10	1.434 (4)	C5′—C6′	1.374 (5)
C5—O5	1.352 (4)	C11—C12	1.474 (5)
C5—C6	1.381 (4)	C12—C13	1.355 (5)
C5—C10	1.412 (4)	C13—C15	1.451 (6)
C6—C7	1.388 (4)	C13—C14	1.475 (6)
C7—O7	1.362 (4)	O7—C16	1.476 (4)
C7—C8	1.384 (4)	C16—C17	1.490 (5)
C8—C9	1.391 (4)	C16—C20	1.508 (6)
C8—C18	1.455 (4)	C16—C19	1.508 (5)
C9—C10	1.395 (4)	C17—C18	1.316 (5)

C9—O1—C2	120.6 (2)	C6′—C1′—C2′	118.0 (3)
C3—C2—O1	122.9 (3)	C6′—C1′—C2	119.6 (3)
C3—C2—C1′	127.0 (3)	C2′—C1′—C2	122.4 (3)
O1—C2—C1′	110.1 (2)	O2′—C2′—C3′	115.9 (3)
C2—C3—C4	119.3 (3)	O2′—C2′—C1′	123.5 (3)
C2—C3—C11	121.3 (3)	C3′—C2′—C1′	120.6 (3)
C4—C3—C11	119.1 (3)	C2′—C3′—C4′	119.8 (3)
O4—C4—C10	120.8 (3)	O4′—C4′—C3′	122.1 (3)
O4—C4—C3	122.1 (3)	O4′—C4′—C5′	117.1 (3)
C10—C4—C3	117.1 (3)	C3′—C4′—C5′	120.8 (3)
O5—C5—C6	119.0 (3)	C4′—C5′—C6′	119.2 (3)
O5—C5—C10	120.3 (3)	C5′—C6′—C1′	121.5 (3)
C6—C5—C10	120.7 (3)	C12—C11—C3	115.5 (3)
C7—C6—C5	119.0 (3)	C13—C12—C11	125.5 (4)
O7—C7—C8	120.2 (3)	C12—C13—C15	125.3 (4)
O7—C7—C6	116.7 (3)	C12—C13—C14	119.8 (4)
C8—C7—C6	123.0 (3)	C15—C13—C14	114.9 (4)
C7—C8—C9	116.3 (3)	C7—O7—C16	118.0 (2)
C7—C8—C18	118.7 (3)	O7—C16—C17	110.0 (3)
C9—C8—C18	125.0 (3)	O7—C16—C20	107.3 (3)
O1—C9—C10	120.5 (2)	C17—C16—C20	110.5 (3)
O1—C9—C8	116.1 (2)	O7—C16—C19	103.6 (3)
C10—C9—C8	123.5 (3)	C17—C16—C19	112.2 (3)
C9—C10—C5	117.4 (3)	C20—C16—C19	112.8 (4)
C9—C10—C4	119.5 (3)	C18—C17—C16	121.4 (3)
C5—C10—C4	123.1 (3)	C17—C18—C8	119.4 (3)

Compound (II)

Crystal data

$C_{27}H_{28}O_6$
M_r = 448.49
Triclinic
$P\bar{1}$
a = 11.326 (2) Å
b = 11.806 (2) Å
c = 9.688 (2) Å
α = 99.87 (2)°
β = 103.31 (2)°
γ = 105.73 (1)°
V = 1175.0 (4) Å^3
Z = 2
D_x = 1.268 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.54180 Å
Cell parameters from 25 reflections
θ = 59–60°
μ = 0.727 mm^{-1}
T = 293 (2) K
Plate
0.4 × 0.4 × 0.3 mm
Orange

Data collection

Rigaku AFC-6 four-circle diffractometer
Profile data from ω–2θ scans
Absorption correction: none
3712 measured reflections
3712 independent reflections
3265 observed reflections [$I > 2\sigma(I)$]

θ_{max} = 62.49°
h = −12 → 13
k = −13 → 13
l = −11 → 10
3 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0495
$wR(F^2)$ = 0.1422
S = 0.693
3712 reflections
321 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1507P)^2 + 0.5679P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = −0.032
$\Delta\rho_{max}$ = 0.242 e Å^{-3}
$\Delta\rho_{min}$ = −0.380 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.86515 (10)	0.73563 (10)	0.60875 (12)	0.0454 (3)
C2	0.9886 (2)	0.74042 (14)	0.6719 (2)	0.0448 (4)
C3	1.0585 (2)	0.69261 (15)	0.5988 (2)	0.0470 (4)
C4	1.0044 (2)	0.63754 (15)	0.4426 (2)	0.0479 (4)
O4	1.06682 (13)	0.59520 (13)	0.3679 (2)	0.0649 (4)
C5	0.8107 (2)	0.58130 (15)	0.2257 (2)	0.0466 (4)
O5	0.87243 (13)	0.53502 (13)	0.13813 (14)	0.0600 (4)
C6	0.6862 (2)	0.5762 (2)	0.1679 (2)	0.0480 (4)
C7	0.6230 (2)	0.62290 (15)	0.2584 (2)	0.0441 (4)
C8	0.6818 (2)	0.67796 (14)	0.4079 (2)	0.0429 (4)
C9	0.8077 (2)	0.68113 (14)	0.4626 (2)	0.0412 (4)
C10	0.8750 (2)	0.63400 (14)	0.3770 (2)	0.0436 (4)
C1′	1.0336 (2)	0.80509 (15)	0.8292 (2)	0.0451 (4)
C2′	1.10261 (15)	0.92985 (15)	0.8725 (2)	0.0445 (4)
O2′	1.11674 (13)	0.98447 (11)	0.76139 (13)	0.0567 (4)
C3′	1.1500 (2)	0.9898 (2)	1.0187 (2)	0.0479 (4)
C4′	1.1282 (2)	0.9268 (2)	1.1239 (2)	0.0491 (4)
O4′	1.17954 (14)	0.99620 (13)	1.26450 (14)	0.0647 (4)
C5′	1.0596 (2)	0.8044 (2)	1.0839 (2)	0.0552 (5)
C6′	1.0125 (2)	0.7451 (2)	0.9360 (2)	0.0535 (5)
C7′	1.2027 (2)	1.1046 (2)	0.7989 (3)	0.0737 (6)
C8′	1.1596 (3)	0.9401 (3)	1.3790 (2)	0.0875 (8)
O7	0.50207 (11)	0.62058 (12)	0.19378 (12)	0.0543 (3)
C11	1.1924 (2)	0.6973 (2)	0.6742 (2)	0.0560 (5)
C12	1.2939 (2)	0.8154 (2)	0.6916 (2)	0.0552 (5)
C13	1.4149 (2)	0.8469 (2)	0.7652 (2)	0.0604 (5)
C14	1.5087 (2)	0.9670 (2)	0.7747 (4)	0.0938 (9)
C15	1.4704 (2)	0.7647 (3)	0.8408 (4)	0.1028 (10)
C16	0.4149 (2)	0.6295 (2)	0.2837 (2)	0.0511 (4)
C17	0.4882 (2)	0.7143 (2)	0.4319 (2)	0.0592 (5)
C18	0.6120 (2)	0.7340 (2)	0.4911 (2)	0.0528 (5)
C19	0.3520 (2)	0.5038 (2)	0.2980 (3)	0.0753 (6)
C20	0.3187 (2)	0.6779 (2)	0.1986 (2)	0.0676 (6)

Table 4. *Selected geometric parameters (Å, °) for (II)*

O1—C2	1.373 (2)	C1′—C2′	1.403 (2)
O1—C9	1.370 (2)	C2′—O2′	1.364 (2)
C2—C3	1.345 (3)	C2′—C3′	1.377 (2)
C2—C1′	1.482 (2)	O2′—C7′	1.415 (2)
C3—C4	1.452 (3)	C3′—C4′	1.388 (3)
C3—C11	1.506 (2)	C4′—O4′	1.364 (2)
C4—O4	1.256 (2)	C4′—C5′	1.379 (3)
C4—C10	1.444 (2)	O4′—C8′	1.416 (3)
C5—O5	1.353 (2)	C5′—C6′	1.389 (3)
C5—C6	1.372 (3)	O7—C16	1.471 (2)
C5—C10	1.420 (2)	C11—C12	1.502 (3)
C6—C7	1.386 (2)	C12—C13	1.307 (3)
C7—O7	1.359 (2)	C13—C14	1.497 (3)
C7—C8	1.401 (2)	C13—C15	1.496 (3)
C8—C9	1.390 (2)	C16—C17	1.503 (2)
C8—C18	1.451 (3)	C16—C19	1.507 (3)
C9—C10	1.393 (2)	C16—C20	1.510 (3)
C1′—C6′	1.379 (3)	C17—C18	1.325 (3)
C2—O1—C9	119.6 (1)	C6′—C1′—C2	121.7 (2)
C3—C2—O1	123.7 (2)	C2′—C1′—C2	119.8 (2)
C3—C2—C1′	125.1 (2)	O2′—C2′—C3′	124.1 (2)
O1—C2—C1′	111.1 (1)	O2′—C2′—C1′	115.6 (2)
C2—C3—C4	119.1 (2)	C3′—C2′—C1′	120.3 (2)
C2—C3—C11	121.7 (2)	C2′—O2′—C7′	117.7 (2)
C4—C3—C11	119.2 (2)	C2′—C3′—C4′	119.8 (2)
O4—C4—C10	121.5 (2)	O4′—C4′—C5′	124.9 (2)
O4—C4—C3	121.9 (2)	O4′—C4′—C3′	114.3 (2)
C10—C4—C3	116.6 (2)	C5′—C4′—C3′	120.8 (2)
O5—C5—C6	119.8 (2)	C4′—O4′—C8′	118.2 (2)
O5—C5—C10	119.7 (2)	C4′—C5′—C6′	118.7 (2)
C6—C5—C10	120.6 (2)	C5′—C6′—C1′	121.8 (2)
C5—C6—C7	119.5 (2)	C7—O7—C16	119.4 (1)
O7—C7—C6	116.9 (2)	C12—C11—C3	114.1 (2)
O7—C7—C8	120.3 (2)	C13—C12—C11	126.1 (2)

C6—C7—C8	122.6 (2)	C12—C13—C14	121.9 (2)
C9—C8—C7	116.2 (2)	C12—C13—C15	122.7 (2)
C9—C8—C18	125.0 (2)	C14—C13—C15	115.4 (2)
C7—C8—C18	118.7 (2)	O7—C16—C17	110.2 (1)
O1—C9—C8	116.0 (2)	O7—C16—C19	108.0 (2)
O1—C9—C10	120.5 (2)	C17—C16—C19	110.7 (2)
C8—C9—C10	123.5 (2)	O7—C16—C20	104.7 (2)
C9—C10—C5	117.6 (2)	C17—C16—C20	111.5 (2)
C9—C10—C4	120.3 (2)	C19—C16—C20	111.5 (2)
C5—C10—C4	122.1 (2)	C18—C17—C16	122.1 (2)
C6′—C1′—C2′	118.5 (2)	C17—C18—C8	119.5 (2)

For both compounds, data collection: *MSC/AFC* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structures: *SHELXL*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1170). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Benzoyl-3-(4-nitrophenyl)thiourea

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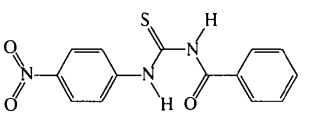
Abstract

In the molecules of the title compound, $C_{14}H_{11}N_3O_3S$, there is an intramolecular N—H···O hydrogen bond [2.635 (3) Å] between the amide N and benzoyl O atoms which completes a nearly planar six-membered ring in the central part of the molecule. The benzene rings of the benzoyl and nitrophenyl moieties form angles of 30.5 (4) and 35.7 (4)°, respectively, with the plane of this hydrogen-bonded ring. In the crystal, molecules are connected into infinite zigzag chains by N—H···O bonds and these chains are linked across centres of symmetry by weak N—H···S interactions, thus forming a two-dimensional network. Van der Waals interactions between layers lead to a crystal structure with one very short axis (4 Å).

Comment

During our systematic search for non-linear optical organic crystals having a short cut-off wavelength, we isolated the title compound, (I).



(I)

Since we do not have access to the Cambridge Structural Database (Allen *et al.*, 1979), a literature search was carried out on compounds of the type $R^1C_6H_4$–CO–NH–CS–NH–$C_6H_4R^2$. The structure determination of one similar compound was found with R^1 = Cl and R^2 = H (Dago, Simonov, Pobedimskaya, Martin & Masias, 1988), which has bond lengths and angles in close agreement with those of the present determination. An N(2)—H···O(1) intramolecular hydrogen bond [2.635 (3) Å] completes an almost planar six-membered ring with atoms C(1), N(1) and C(8); the maximum deviation from the best plane of the five non-H atoms is 0.014 (2) Å and the S atom is 0.104 (2) Å out of the plane (Fig. 1). The NO_2 group is twisted by 10.0 (4)° from the plane of the benzene ring to which it is attached.